

Claims :

1. An integrated process for extracting and purifying tocotrienols/tocopherols, carotenoids and sterols and production of fatty acid esters from oils, comprising the steps of:

- 5 a. Trans-esterification of oil containing tocotrienols/tocopherols, carotenes, sterols, fatty acids, mono-, di- and triglycerides, for a period of time at specific temperature in the presence of a monohydric alcohol, and base or acid to form an ester-rich layer and a glycerol-rich layer;
- 10 b. Separating the ester-rich layer from the glycerol-rich layer as obtained in (a) by gravitational settling, decantation or separation by centrifugal forces;
- c. Washing and drying the ester-rich layer obtained in (b) under conditions sufficient to remove all impurities and base or acid
- 15 without destroying the tocotrienols, tocopherols and carotenoids in the ester-rich layer;
- d. Step-wise molecular distillation or any other distillation of the resultant dried ester-rich layer as obtained in (c) to yield a concentrated mixture of tocotrienols/tocopherols, carotenoids and
- 20 sterols at specific temperature and pressure;
- e. Further trans-esterification of the mixture obtained in (d) containing concentrated tocotrienols/tocopherols, carotenes, sterols, and fatty acids, mono-, di- tri glycerides, for a period of time at specific temperature in the presence of a monohydric alcohol, and base or
- 25 acid to convert glycerides in the oil to form an ester-super-rich layer and a glycerol-rich layer; and
- f. Repeating the above trans-esterification reactions and step-wise molecular distillations to achieve the desired concentration of tocotrienols/tocopherols, carotenoids and sterols.

- 2 The process according to Claim 1, wherein the concentrated carotenoids are treated with a lower alkyl alcohol under conditions sufficient to form carotenoids miscelles without destroying the carotenoids, thereby forming a carotenoid-rich layer.
- 5 3. The carotenoid-rich layer according to Claim 2, wherein the said carotenoid-rich layer is subjected to an evaporation or distillation process to distill out the lower alkyl alcohol to form a concentrated carotenoid extract.
- 10 4. The process according to Claim 1, wherein the concentrated tocotrienols/tocopherols/sterols mixture is treated with a low monohydric alcohol for a period of time at specific temperature to crystallize out the sterols and mono-, di-, and tri-glycerides from the mixture.
- 15 5. The concentrated tocotrienols/tocopherols/sterols mixture according to Claim 4, wherein the said mixture is subjected to a solid-liquid filtration to yield a rich tocotrienols/tocopherols filtrate and sterols cake.
- 20 6. The tocotrienols/tocopherols filtrate according to Claim 5, wherein the said filtrate is subjected to an evaporation or distillation process to distill out the lower alkyl alcohol to form a concentrated tocotrienols/tocopherols extract.
- 25 7. The sterols cake according to Claim 5, wherein the sterols cake is treated with appropriate solvents for a period of time at specific temperature to concentrate the sterols.
8. The concentrated tocotrienols/tocopherols extract obtained from the crystallization process as claimed in Claim 4, wherein the said tocotrienols/tocopherols concentrate is treated with appropriate solvents and absorbents, bleaching earth or activated carbon for a period of time at specific temperature to reduce the colour, and to obtain a lighter coloured tocotrienols/tocopherols concentrate.

9. The process according to Claim 1, wherein the oil used in the transesterification is selected from but not limited to, crude palm oil, crude palm olein, red palm oil, red palm olein, vegetable oil or any other suitable edible oil.
- 5 10. The process according to Claim 1, wherein glycerides in the oil are converted to fatty acid alkyl esters and glycerol, and to form an ester-rich layer and a glycerol-rich layer, by contacting the oil with an esterification solution comprising lower alkyl alcohol and a base or acid.
- 10 11. The process according to Claim 1, wherein the ratio of oil to the esterification solution is in the range between 0.5 - 10 part of oil to 1 part esterification solution.
12. The process according to Claim 1, wherein the base used in the esterification solution is selected from a group comprising of but not limited to sodium methoxide, potassium methoxide, sodium hydroxide, potassium hydroxide, or any other suitable base.
- 15 13. The process according to Claim 1 or 12, wherein the ratio of base to lower alkyl alcohol in the esterification solution is in the range between 0.005 to 5 part of base to 1 part of lower alkyl alcohol.
- 20 14. The process according to Claim 1, wherein the acid used is selected from a group comprising of but not limited to, hydrochloric acid, phosphoric acid, citric acid or any other suitable acid.
15. The process according to Claim 1 or 14, wherein the ratio of acid to lower alkyl alcohol in the esterification solution is in the range between 0.005 to 5 part of acid to 1 part of lower alkyl alcohol.
- 25 16. The process according to Claim 1, wherein the lower alkyl alcohol used is selected from the group comprising of but not limited to methanol, ethanol, butanol, propanol or any other suitable lower alkyl alcohol.

17. The process according to Claim 1, wherein the trans-esterification is carried out at temperature ranging from 5°C to 90°C with time period ranging from 0.50 hour to 16 hours.
- 5 18. The process according to Claim 1, wherein the trans-esterification mixture is agitated at a speed of between 10 rpm to 500 rpm.
19. The process according to Claim 1, wherein the alkyl esters produced comprise methyl, ethyl, and isopropyl or butyl esters of the fatty acids, depending on the type of lower alkyl alcohol used.
- 10 20. The process according to Claim 1, wherein the ester-rich layer or ester-super-rich layer is separated from glycerol-rich layer by conventional gravitational settling or centrifugal forces.
- 15 21. The process according to Claim 1, wherein the ester-rich layer or ester-super-rich layer is washed with either hot or cold water via direct contact with the hot water or through a counter-current hot water column at a temperature ranging between 30 to 90°C.
22. The process according to Claim 1, wherein the ester-rich layer or ester-super-rich layer is washed with hot water till a pH of 6 to 8 is reached.
- 20 23. The process according to Claim 1, wherein the washed ester-rich layer or ester-super-rich layer is subjected to vacuum evaporation or wiped film evaporator or short path distillation to achieve a moisture content of between 0.001% to 0.20%.
- 25 24. The process according to Claim 1, wherein the dried ester-rich layer or ester-super-rich layer is subjected to a multi-stage molecular distillation at temperature ranging from of 50°C to 300°C and at vacuum of 0.00001 to 1.0 mbar.

25. The process according to Claim 1, wherein the mixture obtained in 1(d) comprises a concentrated mixture of tocotrienols/tocopherols, carotenoids and sterols at concentration of between 0.1-10%, 0.1-10% and 0.1-10% respectively.
- 5 26. The process according to Claim 1, wherein the multi-stage molecular distillation of the dried ester-super-rich layer will produce tocotrienols/tocopherols/sterols extract as the distillate and carotenoids extract as the residue.
- 10 27. The process according to Claim 1 or 26, wherein the content of tocotrienols/tocopherols/sterols in the distillate is 5% to 30% total tocotrienols/tocopherols and 5% -50% total sterols and carotenoids content in the residue is between 5% - 30%.
28. The process according to Claim 2 or 3, wherein the lower alkyl alcohol used in alcoholic washing is selected from the group consisting of but not limited to, methanol or ethanol or propanol or butanol or isopropyl alcohol or any combination of these alkyl alcohols.
- 15 29. The process according to Claim 2 or 3, wherein the washing and agitation time ranges from half an hour to 30 hours and the temperature ranging from between 5°C to 90°C.
30. The process according to Claim 2 or 3, wherein the concentrated carotenoids extract has a concentration of between 20%-50% total carotenoids.
- 20 31. The process according to Claim 2 or 3, wherein the concentrated carotenoids extract consists of alpha-carotene and beta-carotene as the major carotenoids and other carotenoids such as gamma-carotene, lycopene, phytoene and phytofluene at lower concentration.

32. The process according to any one of Claims 4 to 6, wherein the lower alkyl alcohol used in the crystallization of tocotrienols/tocopherols/sterols mixture is selected from the group consisting of but not limited to methanol or ethanol or propanol or butanol or any combination of these alkyl alcohols.
- 5 33. The process according to any one of Claims 4 to 6, wherein the crystallization temperature ranges from - 60°C to 0°C for a period ranging from 3 hours to 10 days.
34. The process according to any one of Claims 4 to 6, wherein the evaporation temperature ranges from 10°C to 90°C.
- 10 35. The process according to any one of Claims 4 to 6, wherein the resulting tocots concentrate has a total concentration of tocotrienols and tocopherols ranging from between 20% to 90%
- 15 36. The process according to any one of Claims 4 to 6, wherein the resulting tocots concentrate comprise all eight forms of vitamin E, namely, alpha-tocopherol, beta-tocopherol, gamma-tocopherol, delta-tocopherol and alpha-tocotrienol, beta-tocotrienol, gamma-tocotrienol and delta-tocotrienol.
- 20 37. The process according to any one of Claims 4 to 6, wherein the resulting tocots concentrate may also contain other compounds such as squalene, sterols, carotenoids and CoQ10 with typical concentration ranging between 0.5%-20%, 0.5% - 20%, 0.05% - 10% and 0.001% - 2% respectively.
38. The process according to Claim 5 or 7, wherein the solvent used in the purification of sterols is selected from the group consisting of but not limited to hexane, heptane, iso-octane, acetone or ethyl acetate or any combination of these solvents, in the ratio ranging from between 1:1-10:1.

39. The process according to Claim 5 or 7, wherein the crystallization of sterols is carried out at a temperature ranging from -30°C to 10°C for 12 to 72 hours and the resulting filtered and dried sterols have a total phytosterols content ranging from 70% to 90%.
- 5 40. The process according to Claim 5 or 7, wherein the temperature range of the purification of sterols is between 10°C to 80°C and the period ranging from 1 to 10 hours.
- 10 41. The process according to Claim 8, wherein the temperature range of the decolourization process is between 10°C to 90°C and the period ranging from 1 to 24 hours per batch.
42. The process according to Claim 8, wherein the mixture is agitated in the range from 10 rpm to 1000 rpm.
- 15 43. The process according to Claim 8, wherein the mixture after reaction is filtered with filter press or vacuum filtration or centrifugation or simple settling and the resulting filtrate is evaporated at temperature ranging from 10°C to 90°C and a vacuum of between 1 mbar to 0.0001 mbar.
44. The process according to Claim 8, wherein the final decolourized tocopherols concentrate has a colour range of between 1R to 20R when measured with a $5\frac{1}{2}$ inch cell of the Lovibond Tintometer.
- 20 45. Tocotrienols, tocopherols, carotenoids and sterols produced from oils according to the process as claimed in any one of the preceding claims.